The Effects of Pyrolysis Conditions on the Chemical and Physical Properties of Rice Husk Biochar

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Abstract

Our research has identified the location and mineralogy of plant nutrients in rice husk biochar prepared by three procedures, involving pyrolysis at a low temperature (<550ºC). pH, EC, C, N, ash percentage, chemical composition and mineralogy have been measured. Scanning electron microscopy (SEM) analysis determined the morphology and chemical composition of materials in rice husk biochar.

Rice husk biochar is highly alkaline (8.5-9.7) with a high ash content (36-44%) and high silicon, sodium, potassium, calcium and magnesium contents, it contains archerite (KH_2PO_4), calcite ($CaCO_3$), chlorocalcite ($KCaCl_3$), kalicinite ($KHCO_3$), pyrocoproite ($K_2MgO_7P_2$), struvite ($KMgPO_4.6H_2O$), sylvite (KCl) and vaterite ($CaCO_3$). These minerals are at least moderately soluble in water and are sources of plant nutrients when biochar is applied to soil. Mineral grains (sylvite, carbonates and phosphates) are mainly present between relict vascular bundles and parenchyma cells where dissolved elements in xylem and phloem are precipitated during pyrolysis.

Silica, a major constituent of rice husk biochar, is concentrated in outer epidermis cells including protuberances and hairs (trichomes) and also present in the inner epidermis. Rice husk biochar includes a mixture of amorphous and crystalline forms of silica resembling cristobalite and tridymite. It provides a readily soluble form of both lime and plant nutrient elements so its use on acid, infertile soil should be encouraged.

Keywords

Rice Husk Biochar; Slow Pyrolysis; Low Temperature; Chemical Composition

Introduction

Rice is a primary source of food for many people with annual global paddy rice production being about 580 million tonnes. Rice husk, a by-product of rice processing with approximately 140 million tonnes produced each year, constitutes a significant waste disposal problem (Tarley and Arruda, 2004; Kalderis et al., 2008). Thailand produces around 21-26 million tonnes of rice per year creating about 5.4 million tonnes of rice husks (OAE, 2003). Farmers use rice husk as fuel and for pyrolysis producing bio-oil, gases and biochar. Application of biochar to soil may have diverse agronomic benefits and may also be eligible for carbon credits (Sohi, et al., 2010). Biochar contains plant nutrient elements but the amounts, speciation and availability to plants of these elements is poorly understood, which is the subject of this paper.

The conditions imposed under controlled pyrolysis affect biochar properties. Pyrolysis temperature is the most significant process parameter with the carbon content of biochar created at different temperatures being inversely related to biochar yield (Okimori, et al., 2003). It has been suggested that biochar created at low temperature may be most suitable for controlling the release of plant nutrients from biochar (Day et al., 2005). In the present work, biochar was prepared from rice husks at a low temperature (<550ºC) under laboratory conditions and also under conditions employed by farmers.

Pyrolysis was performed at Kasetsart University (fixed

bed reactor) and the Land Development Department (cylinder reactor) at 400-550°C which is close to the temperature of the traditional kiln (~350°C). The aim of this work was to characterize rice husk biochar, including morphology, mineralogy and chemical composition to identify if the method of manufacture affects these properties.

Materials and Methods

Manufacture of Rice Husk Biochars

A bulk rice husk sample was obtained from a commercial rice mill that processes jasmine rice. Pyrolysis was carried out using KU (Kasetsart University) and LDD (Land Development Department) equipment and in a traditional farmers kiln under limited oxygen atmosphere as shown in Fig. 1. The KU reactor produced biochar from a load of 700 cm³ of biomass at 400-550°C, 10-20°C/min heating rate and N_2 was added into the reactor at 200 cm³/min flow rate with a 1 hr residence time (Fig. 1a). The LDD reactor produced biochar at 400°C with a 300 cm³/min N_2 flow rate (Fig. 1b) from a load of 5,000 cm³ biomass. Biochar was produced in a traditional farmer kiln shown in Fig. 1c where organic waste fuel is burnt in an outer metal cylinder to heat the rice husks in the inner cylinder from which air is excluded. The operating temperature is approximately 350°C with a load of 2,500 cm³ biomass (Figu. 1c).
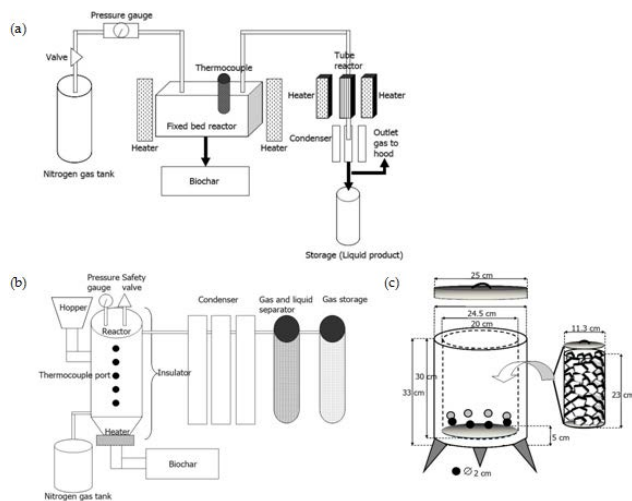

FIG. 1 EQUIPMENT USED TO MANUFACTURE BIOCHAR (a) KASETSART UNIVERSITY EQUIPMENT (KU REACTOR, 370-550°C WITH 200 CM³ MIN⁻¹ N_2 FLOW RATE), (b) LAND DEVELOPMENT DEPARTMENT EQUIPMENT (LDD REACTOR, 400°C WITH 300 CM³/MIN N_2 FLOW RATE), AND (c) TRADITIONAL KILN (UNCONTROLLED TEMPERATURES ~350°C WITH LIMITED OXYGEN).

X-ray Diffraction Analysis

The mineral composition of ground rice husk biochar was determined by conventional X-ray diffraction (CXRD) analysis using a Philips PW-3020 diffractometer with a graphite diffracted beam monochromator (CuKα, 50 kV, 20 mA) scanning from 4 to 70° 2θ, using a step size of 0.02° 2θ and a scan speed of 0.04° 2θ sec⁻¹.

Thermal Analysis

Thermogravimetric data were obtained with a Perkin Elmer STA 6000 with air flowing at 20 mL/min.. Ground rice husk biochar powder was mixed with aluminium oxide (1:1, v/v) before analysis to avoid artefacts in data due to shrinkage of samples during combustion The temperature was raised from 30°C to 100°C at 10°C/min then kept at 100°C for about 10 mins. Then the temperature was increased from 100°C to 1000°C at a 10°C/min heating rate while DTA and TGA data were recorded.

Chemical Analysis

Rice husk biochars were ground in a pestle and mortar for chemical and thermal analysis. Biochar pH and electrical conductivity (EC) were measured using a 1:5 solid: MilliQ (MQ) water (by volume) extraction with shaking end-over-end for 12 hr. The total water soluble elements in biochar were determined by extracting the biochar with MQ water (0.3 g biochar per 10 mL MQ water), shaking end-over-end for 12 hr, and the extracted solutions were analysed by ICP-OES. The ash content of biochars was determined by dry combustion in a ventilated muffle furnace at 600°C overnight. Total carbon and nitrogen concentrations were determined using a Vario Macro elemental analyzer. Total element concentrations were determined by digesting biochar ash in 10% HCl, with analysis of the solutions by ICP-OES (Perkin-Elmer).

Microanalysis

For observation of biochar morphology with a scanning electron microscope and element mapping using EDS (energy dispersive spectroscopy), fractured rice husk biochar particles were placed on a double-sided carbon tape, adhered to an aluminium stub and were coated with carbon. The samples were observed with a JEOL 6400 SEM operated at an acceleration voltage of 15 kV. SEM images of biochar enabled recognition of plant organs that have been altered to assemblages of carbon and mineral grains. EDS provided quantitative analyses of micronsize volumes enabling the determination of the chemical composition of mineral grains on the surface and inside voids in biochar.

Results and Discussions

Mineralogical Properties

The mineralogical composition and the solubility in water of the mineral constituents of rice husk biochars are shown in Table 1 and Fig. 2. Amorphous (noncrystalline) silica, a major constituent of these biochars, generates a broad XRD peak centred at $2\theta \sim 22.5^\circ$, which can be attributed to the presence of disordered cristobalite (Fig. 2) (Liou, 2004) and amorphous carbon also contributes to this broad peak (Radhika and Sugunan, 2006; Chen et al., 2011).
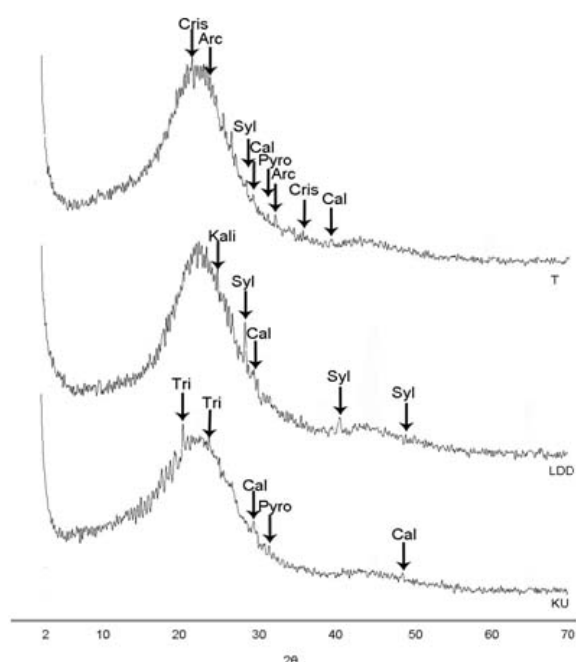

FIG. 2 CONVENTIONAL XRD PATTERNS OF RICE HUSK BIOCHAR (KU=RICE HUSK BIOCHAR FROM KASETSART UNIVERSITY EQUIPMENT, LDD=RICE HUSK BIOCHAR FROM LAND DEVELOPMENT DEPARTMENT EQUIPMENT AND T=TRADITIONAL RICE HUSK BIOCHAR) (ARC=ARCHERITE, CAL=CALCITE, CRI=CRISTOBALITE, KALI = KALICINITE, PYRO=PYROCOPROITE, SYL=SYLVITE, TRI=TRIDYMITE). THE BROAD REFLECTION CENTRED AT ABOUT 22° 2Θ IS DUE TO AMORPHOUS SILICA AND CARBON.

Archerite (KH_2PO_4), calcite ($CaCO_3$), chlorocalcite ($KCaCl_3$), kalicinite ($KHCO_3$), pyrocoproite ($K_2MgO_7P_2$), struvite ($KMgPO_4.6H_2O$), sylvite (KCl) and vaterite ($CaCO_3$) are variously present in rice husk biochar samples. All these minerals are soluble in water to different extents except for cristobalite (SiO_2) and tridymite (SiO_2) (Table 1). It is evident that the solubility in soils of elements in these biochars will depend on the mineral species containing the elements. For example, K in sylvite will readily dissolve when biochar is applied to soil but K in struvite will only be slowly soluble. Calcite ($CaCO_3$), struvite ($KMgPO_4.6H_2O$) and tridymite (SiO_2) are present in all these biochars.

Sylvite (KCl), kalicinite ($KHCO_3$) and vaterite ($CaCO_3$) only occur in LDD biochar. This difference may be a consequence of the different equiptment and procedures used to prepare the biochars.

Thermal Stability

TGA and derivative thermogravimetry DTG curves for the biochars shown in Fig. 3 similar show major exotherm and weight loss at 400-550°C due to oxidation of biochar carbon in the flowing air. The DTGA and DTA curves for LDD biochar have a split DTA/DTGA peak (475°C, 513°C) and an additional peak at 513°C. This difference presumably relates a different (lesser) extent of pyrolysis of the LDD sample.
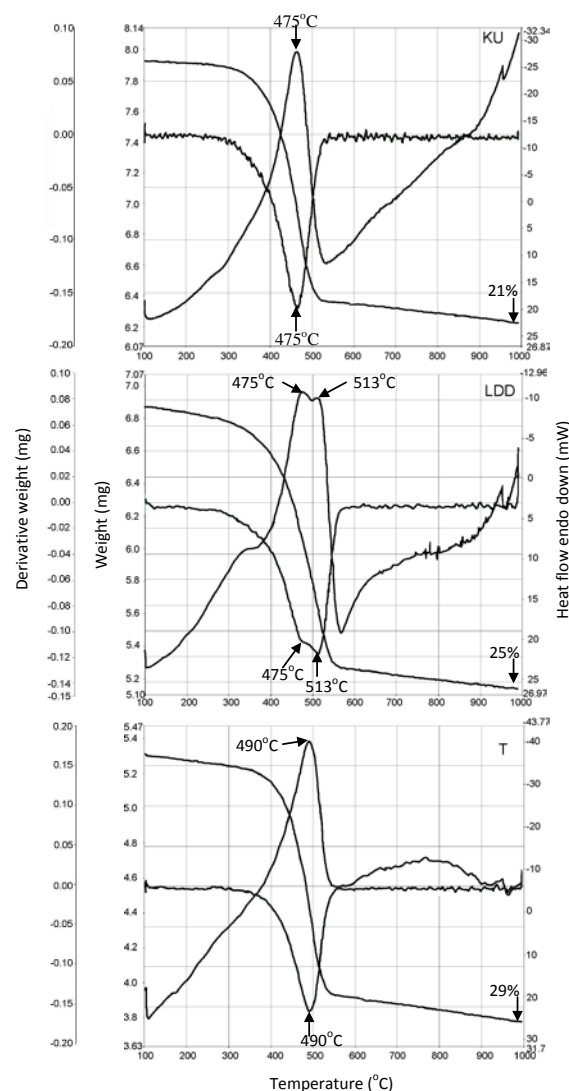

FIG. 3 TGA, DTG AND DTA CURVES FOR RICE HUSK BIOCHAR (KU = RICE HUSK BIOCHAR FROM KASETSART UNIVERSITY EQUIPMENT, LDD = RICE HUSK BIOCHAR FROM LAND DEVELOPMENT DEPARTMENT EQUIPMENT AND T = TRADITIONAL RICE HUSK BIOCHAR). ALUMINIUM OXIDE WAS MIXED WITH BIOCHAR (1:1, V/V). EXOTHERMS AND WEIGHT LOSS ARE DUE TO OXIDATION OF CARBON IN BIOCHAR.

Chemical Properties

Table 2 presents the chemical composition of three rice husk biochars. They have total ash contents ranging between 36-44 wt% and similar carbon contents (46-57%). The higher C content of LDD biochar is consistent with the lower degree of pyrolysis indicated by TGA analysis. EC values for KU and T rice husk biochars are similar but LDD biochar has a much higher value possibly because it contains more of the highly soluble minerals kalicinite, sylvite and vaterite (Table 1).

In addition to, C and N the major constituents of biochars are Si, Ca, Mg, Na, K and P. Other elements are present at minor to trace concentrations. The slightly elevated concentrations of Fe, Cr, Ni and Ti in LDD biochar presumably originated in the iron and steel present in the LDD reactor. The elevated Zn concentration in T biochar may originate in galvanised iron used in this equipment.

The concentration of water soluble elements and the proportion of the total element concentration that is soluble are shown in Tables 3 and 4, respectively. Much K is water soluble because rice husk biochar contains the soluble potassium compounds [archerite (KH_2PO_4), chlorocalcite ($KCaCl_3$), kalicinite ($KHCO_3$), pyrocoproite ($K_2MgO_7P_2$), and sylvite (KCl)]. Potassium in struvite will be only slightly soluble (Table 1). Sulphur is moderately soluble but the forms of S in biochar are not known. Most of the plant nutrient elements in biochar (Ca, Mg, Fe, Mn, Zn, P, S) are quite poorly soluble in water, possibly reflecting the low solubility of minerals at the high pH (8.5-9.7) of the water extract (Table 2).

Anatomical Features and Individual Minerals

Sections of rice husk biochar taken through the entire thickness of the husk provide information on the pseudomorphic preservation of anatomical features for all three methods of biochar manufacture (Fig. 4). Scanning electron microscopy shows that the structure of rice husk biochar faithfully preserves original features of the rice husk. The outer epidermis, layered fibers, vascular bundles, parenchyma cells and inner epidermis and interconnected pores are preserved during the conversion to carbon (Fig. 4). Biochar manufactured by the traditional method (biochar T) has perfectly preserved surface and internal structures (Fig. 4f) whereas rice husk biochar produced on Kasetsart University equipment (KU) has experienced some disruption of the surface and internal structures (Fig. 4b). The porosity provided by the plant cell structure (pore size <10 μm) contributes to the very large specific surface area and high chemical activity of rice husk biochar (Xu et al., 2012). However, much of the surface area is provided by the micropores (< 10 nm pores) in carbon that is not a feature of original husks.

The outer epidermis of rice husk biochars is conspicuously ridged and ridges are punctuated with conical protrusions (Fig. 4). The outer epidermis walls are extremely thick (30-35 μm) (Fig. 4). Hairs are present on the outer epidermis of traditionally prepared rice husk biochar (T) and rice husk biochar manufactured on Land Development Department equipment (LDD) (Fig. 4d, f).

Poorly crystalline silica is a major constituent of rice husk biochar. Much of the silica is present in the outer epidermis cells and concentrated in hair and dome-shaped protrusions (Figs. 5, 6 and 7) and also as dispersed silica inside the husk (Fig. 7). Other minerals (carbonates, phosphates and chlorides) are mainly present between carbonised vascular bundles and parenchyma cells (Figs. 5, 6 and 7). These mineral grains provide x-ray spectra in the SEM that are indicative of individual minerals or mixes of the minerals identified by XRD (Table 1 and Figs. 5, 6 and 7).

TABLE 1 THE COMPOSITION AND SOLUBILITY IN WATER OF MINERAL CONSTITUENTS OCCURRING IN RICE HUSK BIOCHAR. SOLUBILITY VALUES ARE DERIVED FROM THE LITERATURE. THE ABUNDANCE OF THESE MINERALS IN THE THREE BIOCHAR IS INDICATED

Biochars	Formula**	Archerite (KH_2PO_4)	Calcite ($CaCO_3$)	Chlorocalcite ($KCaCl_3$)	Cristobalite (SiO_2)	Kalicinite ($KHCO_3$)	Pyrocoproite ($K_2MgO_7P_2$)	Struvite ($KMgPO_4.6H_2O$)	Sylvite (KCl)	Tridymite (SiO_2)	Vaterite ($CaCO_3$)
	Solubility in water (mg L^{-1})*	Soluble	14	Soluble	Insoluble	224,000	Decomposes	Slightly soluble	238,000	Insoluble	Highly soluble
Rice husk KU		-	x	x	-	-	x	x	-	x	-
Rice husk LDD		x	x	-	-	x	-	x	x	x	xx
Rice husk T		x	x	-	x	-	x	x	-	x	-

KU=biochar from Kasetsart University equipment, LDD = biochar from Land Development Department equipment, T= traditional biochar, - = not detected, x = small, xx = moderate, * Water solubility data from Weast (1983), ** ICDDF (powder data file).

TABLE 2 PROPERTIES AND TOTAL ELEMENTAL COMPOSITION* OF THE RICE HUSK BIOCHARS.

Biochars		Wt loss	Ash	C	N	pH**	EC**	Si	Al	Ca	Mg	Na	K	P	S	Fe	Mn	As	Ba
		(----------%-------------)					(µs cm⁻¹)	(--mg kg⁻¹--)										
Rice husk	KU	62	38	47	0.59	8.5	620	166338	793	2245	900	29628	7999	1033	442	416	249	3	15
Rice husk	LDD	64	36	51	0.91	9.7	3180	149499	500	7804	1840	19086	12519	2162	864	1270	207	nd	63
Rice husk	T	56	44	46	0.63	9.7	833	193748	212	1340	1683	12353	8502	2983	89	220	454	3	23

Biochars		Co	Cr	Cu	Gd	Ge	Ni	Pb	Rb	Sr	Ti	Zn	Zr
		(--mg kg⁻¹--)											
Rice husk	KU	3	9	10	2	5	8	1	18	9	41	31	2
Rice husk	LDD	3	130	24	1	3	19	11	21	18	114	84	7
Rice husk	T	5	4	20	1	6	9	11	78	4	18	309	1

* = digest of biochar ash samples in 10% HCl, measured by ICP-OES and converted to biochar basis, ** = 1:5 H_2O extract on biochar, Wt loss = weight loss on ignition in air at 600°C, nd = not detectable, KU = biochar from Kasetsart University equipment, LDD = biochar from Land Development Department equipment, T = traditional biochar.

TABLE 3 CONCENTRATIONS OF WATER SOLUBLE ELEMENTS* IN RICE HUSK BIOCHAR.

Raw Materials		Al	Na	K	Ca	Mg	P	S	Fe	Mn	Cu	Zn
		(---mg kg⁻¹---)										
Rice husk	KU	0.3	32	1641	59	44	243	73	0.5	9	0.03	0.02
Rice husk	LDD	0.8	710	9903	548	225	129	1118	0.2	0.3	nd	nd
Rice husk	T	0.1	nd	2141	28	341	978	19	0.2	17	nd	0.3

* Biochar samples were extracted by MQ water for 12 hr and analysed by ICP-OES, nd = not detectable, KU = biochar from Kasetsart University equipment, LDD = biochar from Land Development Department equipment, T = traditional biochar.

TABLE 4 THE PROPORTION OF THE TOTAL ELEMENT CONCENTRATION IN RICE HUSK BIOCHAR THAT IS WATER SOLUBLE*.

Raw Materials		Al	Na	K	Ca	Mg	P	S	Fe	Mn	Cu	Zn
Rice husk	KU	0.00	0.00	0.21	0.03	0.05	0.24	0.17	0.00	0.04	0.00	0.00
Rice husk	LDD	0.00	0.04	0.79	0.07	0.12	0.06	>1.00	0.00	0.00	0.00	0.00
Rice husk	T	0.00	0.00	0.25	0.02	0.20	0.33	0.21	0.00	0.04	0.00	0.00

* Biochar samples were extracted by MQ water for 12 hr and analysed by ICP-OES, KU = biochar from Kasetsart University equipment, LDD = biochar from Land Development Department equipment, T = traditional biochar.
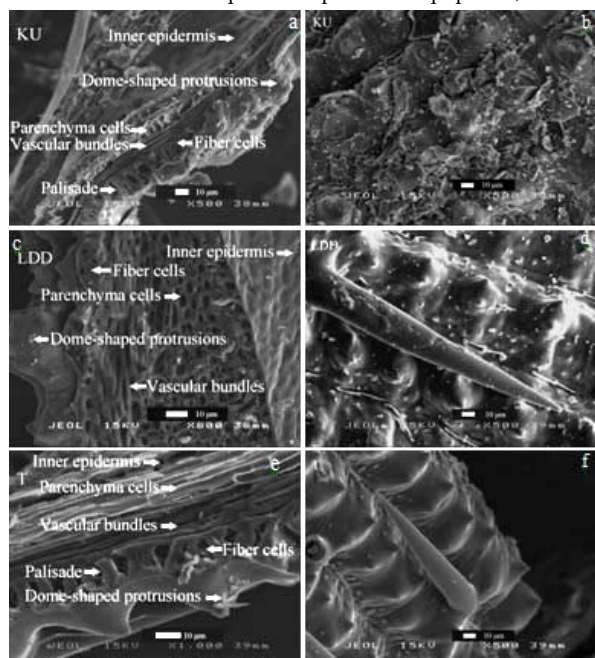

FIG. 4 SCANNING ELECTRON MICROGRAPHS OF (a) RICE HUSK BIOCHAR (b) OUTER EPIDERMIS FROM KASETSART UNIVERSITY EQUIPMENT (KU), (c) RICE HUSK BIOCHAR (d) OUTER EPIDERMIS FROM LAND DEVELOPMENT DEPARTMENT EQUIPMENT (LDD) AND (e) RICE HUSK BIOCHAR (f) OUTER EPIDERMIS FROM THE TRADITIONAL KILN (T). THE WELL PRESERVED RELICT CARBONISED PLANT ORGANS ARE INDICATED.

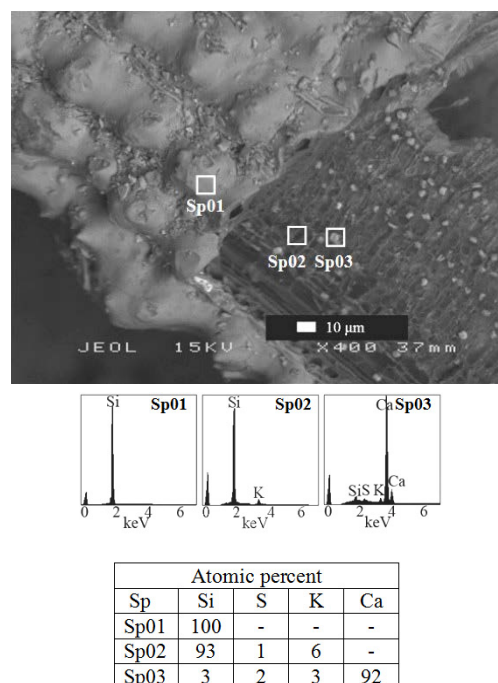| Atomic percent				
Sp	Si	S	K	Ca
Sp01	100	-	-	-
Sp02	93	1	6	-
Sp03	3	2	3	92

FIG. 5 BACKSCATTERED ELECTRON MICROGRAPH, X-RAY SPECTRA AND ANALYSES OF INORGANIC PARTICLES EXPRESSED AS ATOMIC PERCENT FOR RICE HUSK BIOCHAR FROM KASETSART UNIVERSITY EQUIPMENT (KU) (Sp01 = AMORPHOUS SILICA, Sp02 = MOSTLY AMORPHOUS SILICA AND $KHCO_3$ AND Sp03 = MOSTLY $CaCO_3$, LITTLE $KHCO_3$, $CaSO_4$ AND SiO_2)
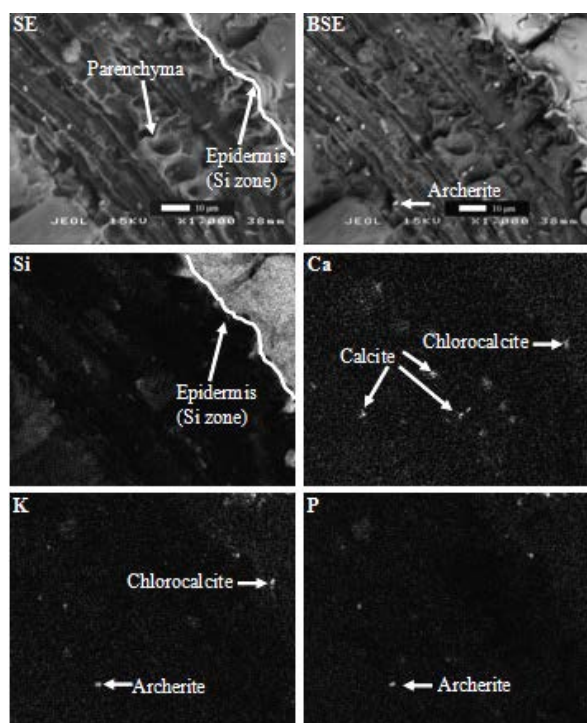
FIG. 6 SECONDARY AND BACKSCATTERED ELECTRON SCANNING MICROGRAPHS AND ELEMENT MAPS FOR RICE HUSK BIOCHAR FROM LAND DEVELOPMENT DEPARTMENT EQUIPMENT (LDD) SHOWING A LAYER OF SILICA AND GRAINS OF CALCITE, ARCHERITE AND CHLOROCALCITE IN THIS SAMPLE.
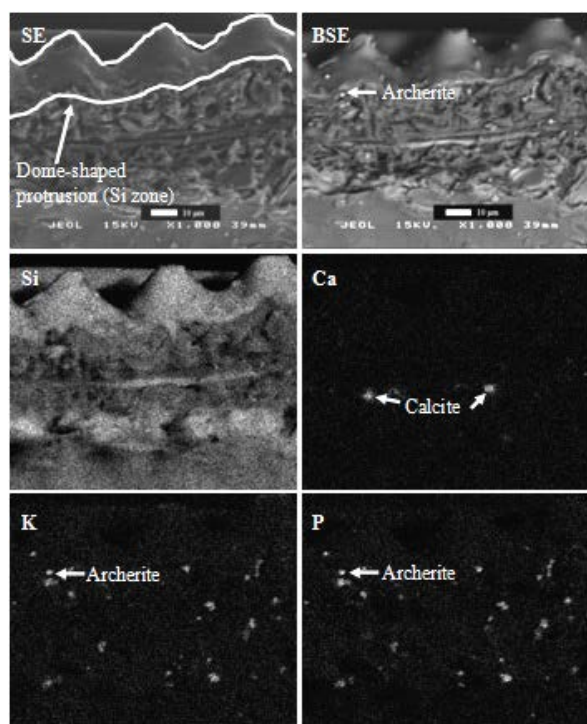

FIG. 7 SCONDARY AND BACKSCATTERED SCANNING ELECTRON MICROGRAPHS AND ELEMENT MAPS FOR TRADITIONAL RICE HUSK BIOCHAR (T) CONTAINING CALCITE AND ARCHERITE.

Conclusions

A aim of this work was to develop an understanding of the speciation of plant nutrient elements in biochar created from rice husks. The bioavailability of elements depends on speciation (mineralogy). Rice husk biochar is rich in total ash ranging between 36-44 wt% and has a high pH (8.5-9.7) due to the presence of alkaline minerals. A broad XRD peak from 15° to 35° 2θ indicates that rice husk biochar samples contains much amorphous silica (Kamath and Proctor, 1998; Tarley et al., 2004) although amorphous carbon also scatters x-rays in this 2θ range. There is no difference in XRD patterns between rice husk biochars produced by the three processes. All rice husk biochar contains the minerals archerite (KH_2PO_4), calcite ($CaCO_3$), chlorocalcite ($KCaCl_3$), kalicinite ($KHCO_3$), pyrocoproite ($K_2MgO_7P_2$), struvite ($KMgPO_4.6H_2O$), sylvite (KCl) and vaterite ($CaCO_3$) all of which contain essential plant nutrients.

Biochar formed from rice husks may sequester carbon in soils while releasing plant nutrients to soil solution as ions diffuse into and out of this highly porous material. The rate at which this process occurs will depend on a numbers of factors including the pore structure of both biochar and soil, the solubility of minerals in biochar and chemical properties including pH of soil and soil solution. This work has clearly demonstrated the value of rice husk biochar as a multinutrient fertilizer.

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